NOR 61-199

DEVELOPMENT OF IMPROVED METHODS, PROCESSES, AND TECHNIQUES FOR PRODUCING STEEL EXTRUSIONS

L. M. Christensen
W. Roser

NORTHROP CORPORATION
Norair Division

Contract AF 33(600)-36713

Interim Engineering Report No. 8
9 March 1961 - 30 June 1961

Dimensional integrity and surface qualities obtained in the .06 tee sections of Phase I are considered sufficient for proceeding to Phase II. During Phase II, additional extruding development is expected to result in further improvement.

BASIC INDUSTRY BRANCH
MANUFACTURING TECHNOLOGY LABORATORY

Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

ABSTRACT - SUMMARY
Interim Technical Progress Report

INTERIM REPORT NOR 61-199
June 1961

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Low and erratic mechanical properties were noted in the evaluation of Phase I extrusions in both H-11 and PH 15-7Mo materials. Metallurgical studies by both Northrop and Armco Steel Corporation indicated that these low properties resulted from diffusion of the nickel lubricant material into the surface of the extrusions.

After removal of the contaminant coating by chemical etching, the resultant mechanical properties were well within specifications for the materials. The chemical etching also yields improved surface quality.

Program planning for Phase II includes efforts to resolve the contamination problem in extruding as well as further improvement of extruding techniques. Cold and warm drawing will then be developed to yield extrusions with wall thicknesses of .04 inch.

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NOTICES

FOREWORD

This Interim Technical Progess Report covers the work performed under Contract AF 33(600)-36713 from 9 March 1961 through 30 June 1961. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with Northrop Corporation, Norair Division, Hawthorne, California was administered under the direction of Mr. T. S. Felker of the Basic Industry Branch (ASRCTB), Manufacturing Technology Laboratory, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

L. M. Christensen of Northrop Norairs' Materials Sciences and Value Analysis Group was the engineer in charge, assisted by Mr. W. Roser. Major subcontractor was Allegheny Ludlum Steel Corporation, Watervliet, New York, with Mr. J. S. Rice in charge of the subcontract engineering effort.

The primary objective of the Air Force Manufacturing Technology Program is to increase producibility, and improve the quality and efficiency of fabrication of aircraft, missiles, and components thereof. This report is being disseminated in order that methods and/or equipment developed may be used throughout industry, thereby reducing costs and giving "MORE AIR FORCE PER DOLLAR".

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Technology development required on this or other subjects will be appreciated.

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PUBLICATION REVIEW

Approved by: R. D. Johnson
Dr. R. D. Johnson, Supervisor
Materials Sciences Laboratory

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF TABLES

1.0 INTRODUCTION

This report is the eighth of a series of Interim Engineering Reports concerning a program for the development of an improved, commercially feasible extruding process to produce ultra thin aircraft quality steel extrusions that are comparable in quality to their aluminum counterparts.

The reporting period covered in this document extends from 9 March 1961, the effective date of the contract revision, through 30 June 1961. The purpose of the contract revision was to include in phases subsequent to Phase I the use of cold and warm drawing to extend the effort to still thinner shapes. Therefore, no formal interim reports were issued during the period in which the contract change was being incorporated.

Included in this report is a complete summary of the results of tests on Phase I extrusions together with a report on the preliminary activities on Phase II.

For the benefit of the reader, Appendix I contains summaries of Interim Engineering Reports which have been published previously on this contract.

2.0 CONTRACT SUMMARY

The following brief summary of the primary contract work statement elements is presented for continuity of this report:

Phase I Survey of the aircraft and extrusion industries to select shapes, alloys, and specifications for the extrusions to be developed in this program.

The three subcontractors chosen to participate in the development of experimental techniques to produce the simplest shape in two selected alloys are:

Allegheny Ludlum Steel Corp., Watervliet, N. Y.
Harvey Aluminum, Torrance, California
C. I. E. P. M., Paris, France

Phase II Development and optimization of cold and warm draw techniques for the .06 thick tee shape which was developed in Phase I.

Phase III Development of both extrusion and draw techniques for an actual B-70 Weapons System structural shape in PH 15-7 Mo steel alloy.

Phase IV Development of both extrusion and draw techniques for the same B-70 shape as Phase III except that the material shall be A-286.

Phase V Development of production methods for heat treatment of steel extrusions.

Allegheny-Ludlum Steel Corporation has been chosen as the subcontractor for the development activity of Phases II - V.

3.0 EVALUATION OF PHASE I EXTRUSIONS

Extrusions from Phase I efforts were inspected for dimensional integrity and surface roughness, and mechanical properties were determined. Results of this evaluation are presented in the following paragraphs.

Substandard results were found in the mechanical property investigation of both PH 15-7 Mo and H-11 extrusions. A lengthy and detailed metallurgical evaluation was performed to determine the causes for these results.

3.1 CONCLUSIONS

1. The dimensional integrity and surface qualities indicated in this report are considered sufficient for proceeding to Phase II; further improvement in surface quality is expected in the extruding development of Phase II.

2. The low, erratic mechanical properties noted in the extruded shapes were due to nickel diffusion into the extrusion surface.

3. The diffusion of nickel does not follow classical theory due to severe surface deformations and/or pressures.

4. The nickel contamination can be removed by chemical etching.

5. Chemical milling utilized to remove the contaminated layer can be quite beneficial to the subsequent drawing operation in that it materially improves the surface quality thus potentially reducing the number of passes.

3.2 DIMENSIONAL INTEGRITY

The leg height, flange width, leg thickness, flange thickness on each side of the leg, and bow determinations were made on each extrusion. Dimensions were checked every 24 inches along the extrusion. The results of these determinations are shown in Tables I and II. Where there are two sets of data for one extrusion, that particular extrusion fractured during straightening. The location of these measurements is shown in Figure 1.

TABLE I

DIMENSIONS OF EXTRUSIONS

H-11 STEEL

Extrusion Number	Length ft-in	Bow Height in.	Dimensions A in.	B in.	C in.	D in.	E in.
2	17-8	0.045	1.486	1.491	0.067	0.066	0.069
			1.490	1.498	0.067	0.066	0.068
			1.493	1.499	0.067	0.066	0.069
			1.493	1.499	0.067	0.066	0.069
			1.495	1.498	0.067	0.067	0.069
			1.482	1.491	0.068	0.067	0.069
			1.488	1.485	0.068	0.068	0.070
			1.492	1.489	0.069	0.068	0.070
			1.510	1.512	0.070	0.070	0.071
			1.515	1.515	0.071	0.071	0.072
4 (1)	5-4	0.025	1.485	1.501	0.059	0.068	0.073
			1.490	1.502	0.056	0.068	0.074
			1.493	1.501	0.056	0.068	0.074
			1.496	1.498	0.057	0.068	0.074
4 (2)	10-9	0.065	1.486	1.486	0.057	0.068	0.072
			1.484	1.487	0.055	0.068	0.072
			1.487	1.487	0.057	0.068	0.072
			1.486	1.486	0.056	0.068	0.073
			1.194	1.486	0.058	0.068	0.073
			1.184	1.498	0.061	0.069	0.074
8	14-6	0.035	1.510	1.522	0.072	0.072	0.072
			1.506	1.516	0.072	0.072	0.072
			1.489	1.492	0.072	0.072	0.071
			1.487	1.494	0.072	0.073	0.072
			1.488	1.457	0.070	0.072	0.072
			1.486	1.443	0.072	0.072	0.074
			1.475	1.423	0.072	0.072	0.074
			1.471	1.401	0.072	0.073	0.074

TABLE I (Cont.)

DIMENSIONS OF EXTRUSIONS

PH 15-7 Mo STEEL

Extrusion Number	Length ft-in	Bow Height in.	Dimensions A in.	B in.	C in.	D in.	E in.
2	13-0	0.130	1.486	1.494	0.070	0.072	0.077
			1.492	1.496	0.068	0.073	0.078
			1.493	1.497	0.069	0.073	0.078
			1.494	1.496	0.069	0.073	0.079
			1.493	1.496	0.069	0.074	0.080
			1.495	1.495	0.070	0.074	0.081
			1.490	1.493	0.070	0.074	0.079
			1.490	1.498	0.071	0.075	0.080
6	20-7	1.168	1.505	1.499	0.070	0.071	0.072
			1.512	1.511	0.069	0.073	0.073
			1.511	1.513	0.069	0.072	0.073
			1.511	1.514	0.068	0.073	0.074
			1.510	1.515	0.068	0.073	0.074
			1.505	1.515	0.068	0.074	0.075
			1.504	1.516	0.067	0.074	0.075
			1.504	1.515	0.068	0.074	0.075
			1.507	1.514	0.069	0.075	0.076
			1.510	1.513	0.070	0.076	0.077
			1.506	1.510	0.070	0.076	0.078
7	10-0	0.025	1.489	1.503	0.067	0.075	0.077
			1.490	1.502	0.069	0.073	0.072
			1.487	1.499	0.068	0.073	0.072
			1.485	1.500	0.066	0.072	0.072
			1.487	1.499	0.067	0.072	0.070
			1.490	1.500	0.064	0.071	0.071
	3-4	0.015	1.439	1.202	0.065	0.077	0.072
			1.463	1.221	0.064	0.073	0.071
			1.473	1.295	0.066	0.074	0.072

TABLE II

SUMMARY OF DIMMENSIONAL

MEASUREMENTS ON EXTRUSIONS

Extrusion Number	Alloy		A in.	B in.	C in.	D in.	E in.
2	H-11	Avg.	1.494	1.498	0.068	0.068	0.070
		Range	1.482-1.515	1.485-1.515	0.067-0.071	0.066-0.071	0.069-0.072
4	H-11	Avg.	1.488*	1.493	0.057	0.068	0.073
		Range	1.184-1.496	1.486-1.502	0.055-0.061	0.068-0.069	0.072-0.074
8	H-11	Avg.	1.489	1.468	0.072	0.072	0.073
		Range	1.471-1.510	1.401-1.522	0.070-0.072	0.072-0.073	0.071-0.074
2	PH 15-7 Mo	Avg.	1.492	1.496	0.070	0.074	0.079
		Range	1.486-1.495	1.493-1.498	0.068-0.071	0.072-0.075	0.077-0.081
6	PH 15-7 Mo	Avg.	1.508	1.512	0.069	0.074	0.075
		Range	1.504-1.512	1.499-1.515	0.068-0.070	0.071 0.076	0.072 0.078
7	PH 15-7 Mo	Avg.	1.478	1.500*	0.066	0.073	0.072
		Range	1.439 1.490	1.202 1.503	0.064 0.069	0.071 0.077	0.070 0.077

* Average excluding dimensions significantly under nominal which would normally be cropped off.

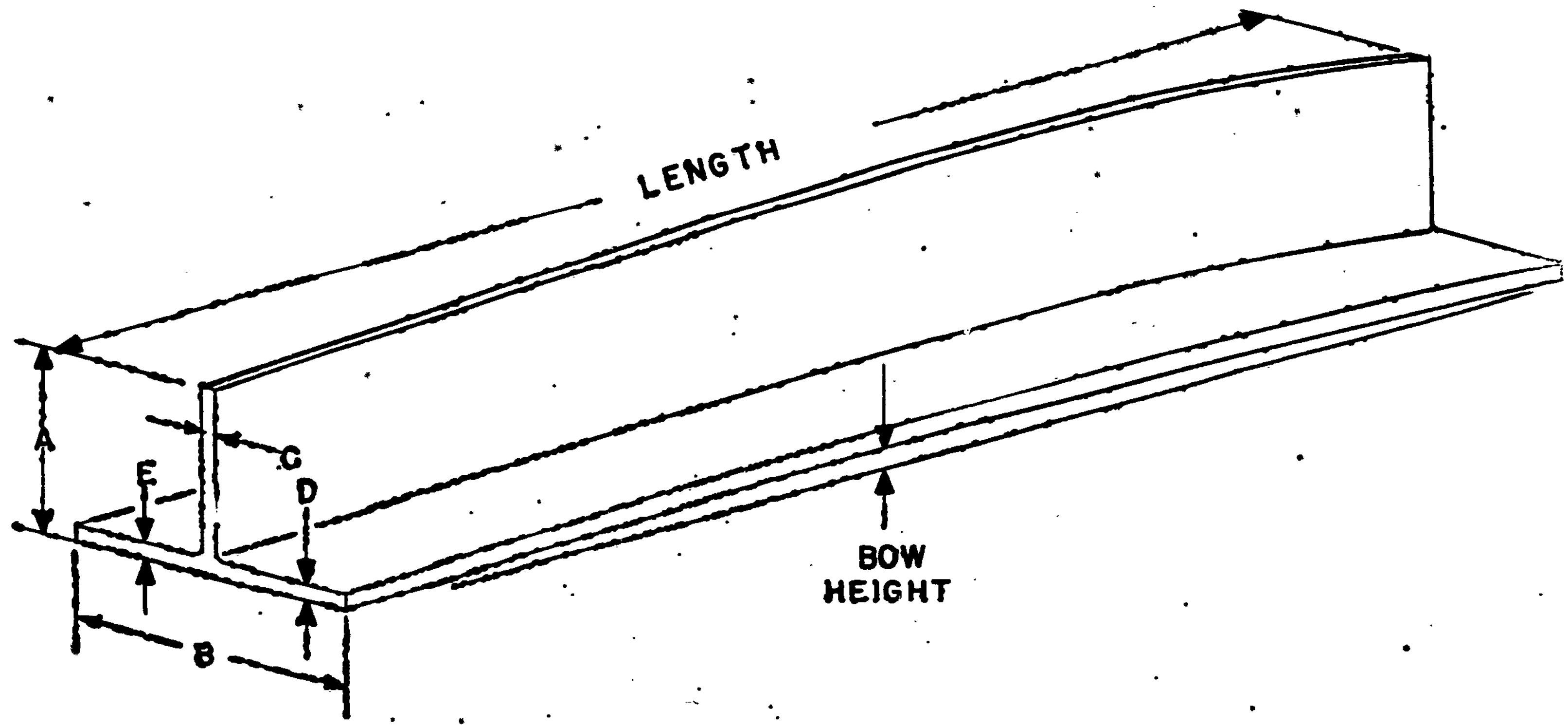

FIGURE 1
LOCATION OF DIMENSIONAL
MEASUREMENTS OF EXTRUSIONS

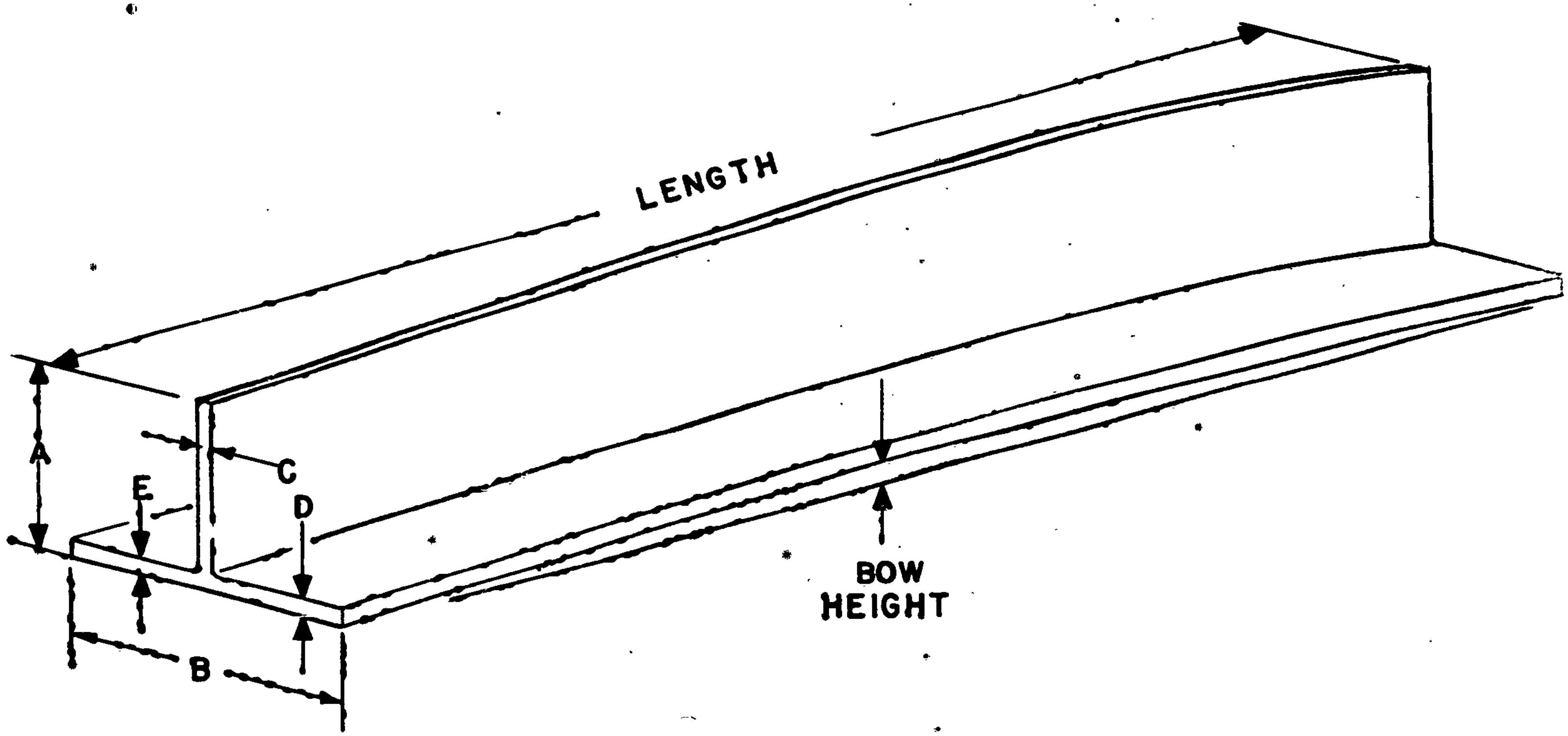

FIGURE 1
LOCATION OF DIMENSIONAL MEASUREMENTS OF EXTRUSIONS

3.3 SURFACE ROUGHNESS

Surface roughness determinations were made at both the front and rear of each extrusion using a Brush Surfindicator. The surface roughness was made both longitudinal and transverse to the extruding direction. As would be anticipated, the roughness increased toward the rear of the extrusion due to die wear. The values of these determinations are shown in Table III. The location and direction in which the roughness measurements were made are shown in Figure 2.

A range of values for each determination is shown rather than an average value. The rather wide range of values reported in some of the areas indicates a local defect.

TABLE III

SURFACE ROUGHNESS

MEASUREMENTS

Extrusion Number	Location	Direction (See Fig. 2) 1 Min	1 Max	2 Min	2 Max	3 Min	3 Max	4 Min	4 Max
		RMS	RMS	RMS	RMS	RMS	RMS	RMS	RMS
2 (PH15-7Mo)	Front	45	50	60	85	45	55	55	65
	Rear	50	55	65	90	60	200	150	200
6 (PH15-7Mo)	Front	55	85	80	90	45	90	45	85
	Rear	50	180	80	95	65	90	60	95
2 (H-11)	Front	30	45	35	70	35	55	55	60
	Rear	60	210	150	210	150	210	110	210
3 (H-11)	Front	45	50	40	55	45	150	50	180
	Rear	45	77	50	80	40	65	65	95
4 (H-11)	Front	210	250	200	240	240	275	250	275
	Rear	300	400	350	400	325	375	275	350
8 (H-11)	Front	45	55	65	75	45	55	65	80
	Rear	55	65	55	150	35	65	60	75

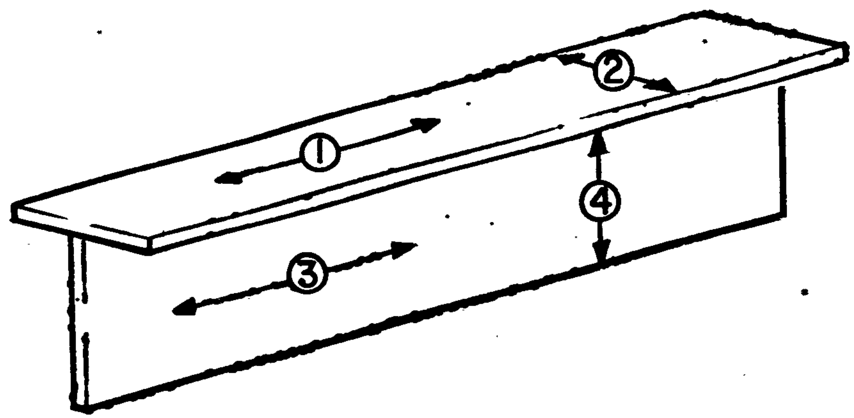

FIGURE 2

SURFACE ROUGHNESS DIRECTIONS

3.4 MECHANICAL PROPERTIES

Tension tests were made on each of the extrusions in the middle and at both ends. Coupons were removed from both the longitudinal and transverse directions and from both the horizontal and vertical members. The results of these tests are shown in Tables IV and V. (See Figure 3 for location of test specimen.) In addition, one PH 15-7 Mo extrusion was submitted to North American Aviation for their evaluation. The results of the North American tests are shown in Table VI.

There is a large difference between the values obtained by North American and those obtained by Norair. So far this difference has not been rectified, but it is being investigated and will be reported in a subsequent report. Extrusion number 7 made from PH 15-7 Mo was tested at a later date than numbers 2 and 6 (PH 15-7 Mo) and specimens were removed only from the ends. The values for this extrusion are shown in Table VII.

TABLE IV

H - 11 STEEL EXTRUSIONS

Extrusion Number	Specimen Number	Yield Strength ksi	Ultimate Strength ksi	Elongation in 2 in. %
2	A-1	200.6	261.4	10.0
	A-2	197.6	253.8	8.5
	A-3	231.2	256.2	8.0*
	A-4	256.0	263.0	10.0*
	B-1	208.2	265.9	10.0
	B-2	194.0	255.6	7.0
	B-3	228-7	252.4	10.0*
	B-4		Void Test	
	C-1	205.9	266.0	10.0
	C-2	196.9	254.9	5.0
	C-3	247.1	255.8	10.0*
	C-4	247.2	261.0	10.0*
3	A-1	247.2	269.2	9.0
	A-2	198.7	264.8	7.0
	A-3	259.7	265.0	10.0*
	A-4	272.3	277.5	10.0*
	B-1	205.9	263.7	10.0
	B-2	196.7	254.8	7.0
	B-3	251.3	264.2	14.0*
	B-4	191.7	207.3	12.0*
	C-1	202.2	259.5	10.0
	C-2	193.2	251.2	9.0
	C-3	258.7	263.7	12.0*
	C-4	269.2	276.9	10.0*

TABLE IV (Cont.)

Extrusion Number	Specimen Number	Yield Strength ksi	Ultimate Strength ksi	Elongation in 2 in. %
4	A-1	200.0	264.8	8.0
	A-2	194.5	244.8	5.0
	A-3	255.7	264.4	10.0*
	A-4	257.0	264.8	8.0*
	B-1	200.0	257.6	8.0
	B-2	194.4	262.1	7.5
	B-3	257.3	266.0	9.0*
	B-4	250.0	264.1	8.0*
	C-1	196.0	257.8	7.5
	C-2	196.3	252.5	7.5
	C-3	258.7	267.4	10.0*
	C-4		No Specimen	
8	A-1	203.0	268.8	8.0
	A-2	192.8	254.4	7.5
	A-3	233.9	257.9	8.0*
	A-4	256.2	272.7	10.0*
	B-1	206.5	259.2	9.5
	B-2	197.0	259.4	6.5
	B-3	251.5	261.4	10.0*
	B-4	259.9	272.3	12.0*
	C-1	208.2	265.8	7.5
	C-2	198.6	257.0	6.5
	C-3	257.8	266.3	12.0*
	C-4	264.0	278.1	12.0*
Required**		215 min.	260 min.	8% min.

* Elongation in 1/2 inch

** AMS 6485

TABLE V

PH 15-7 Mo EXTRUSIONS

Extrusion Number	Specimen Number	Yield Strength ksi	Ultimate Strength ksi	Elongation in 2 In. %
2	A-1	199.5	216.0	7.5
	A-2	193.3	212.0	7.5
	A-3	190.6	199.9	10.0*
	A-4		Test Void	
	B-1	204.2	222.0	9.0
	B-2	199.4	218.0	9.5
	B-3	200.3	211.2	11.0*
	B-4	209.7	213.5	10.0*
	C-1	207.8	225.7	7.0
	C-2	200.6	219.5	9.0
	C-3	212.7	221.5	14.0*
	C-4	212.0	219.8	15.0*
6	A-1	185.3	208.9	10.0
	A-2	172.2	196.1	13.0
	A-3	198.2	211.9	10.0*
	A-4	203.6	216.7	10.0*
	B-1	182.8	207.5	12.5
	B-2	172.7	203.5	15.0
	B-3	206.6	213.0	12.0*
	B-4	191.0	212.4	12.0*
	C-1	198.1	208.7	6.0
	C-2	172.0	196.0	13.0
	C-3	201.5	210.6	10.0*
	C-4	199.0	205.6	11.0*
Required		180 min.	210 min.	5 min.

* Elongation in 1/2 in., transverse specimen

*NAA Material Specification LB 0160-123, "Steel Bar, Rod and Shapes, PH 15-7 Mo, Corrosion Resistant, Precipitation Hardening."

TABLE VI

MECHANICAL PROPERTIES OF PH 15-7 Mo

EXTRUSIONS DETERMINED BY

NORTH AMERICAN AVIATION

ANNEALED COND.

Test Number	Yield Strength ksi	Ultimate Strength ksi	Elongation in 2 in. %
Required*	70.0 max	160.0 max	20% min.
1	61.5	157.7	13.5
2	64.2	160.8	18.5

RH-950 COND.

Test Number	Yield Strength ksi	Ultimate Strength ksi	Elongation in 2 in. %
Required*	180.0 min.	210.0 min	5% min
A-1**	177.2	215.2	11.0
A-2**	182.8	202.9	4.0
B-1	191.4	228.7	6.5
B-2	198.4	232.0	4.0

* NAA Material Specification LB 0160-123, "Steel Bar, Rod and Shapes, PH 15-7 Mo, Corrosion Resistant, Precipitation Hardening."

** No. 1 - Represents the flange member.
No. 2 - Represents the upright member.

TABLE VII

TENSION TEST OF EXTRUSION

No. 7 - PH 15-7 Mo

Specimen Number	Yield Strength ksi	Ultimate Strength ksi	Elongation in 2 in %
7-2-A	166.1	202.5	13.5
7-3-A	196.2*	200.9	11.0**
7-1-A	171.5	209.1	13.0
7-4-A	206.7*	216.7	12.0*
7-2-B	164.0	199.1	13.5
7-3-B	203.8*	209.9	12.0**
7-1-B	176.5	207.7	13.0
7-4-B	202.5*	216.4	12.0**

* Yield Strength calculated from head travel
** Elongation in 1/2 in., transverse specimen

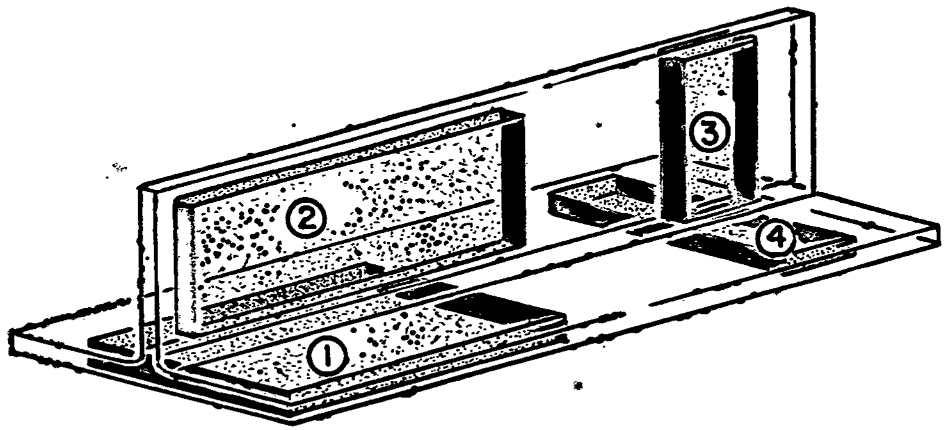

FIGURE 3
SPECIMEN LOCATION OF
TESTS IN TABLES

3.5 HARDNESS SURVEY OF PH 15-7 Mo AND H-11 EXTRUSIONS

The results of mechanical tests indicate rather erratic properties, especially in the yield strength. In order to discover the cause for these low erratic properties, a section of extrusion number 7 made from PH 15-7 Mo was submitted to Armco Steel Corp. for their analysis. A report of their findings is shown in Appendix II. It was the conclusion of Armco that the lowered properties were caused by nickel diffusion from the cladding during the extrusion process resulting in retained austenite being formed at the surface.

An attempt to spectroscopically analyze for depth of nickel contamination in the extrusions was unsuccessful for sundry reasons. Figure 4 is a graph of microhardness surveys made on various PH 15-7 Mo extrusions and in several places in one extrusion. It is apparent that the contamination depth varies from approximately 0.002 in. to 0.007 in. Similar conditions were found in the H-11 extrusions. Figure 5 is a photomicrograph of one of the traverses.

As further test, tensile specimens were machined from extrusion number 6 and 0.01 in. of material was removed from each surface. These specimens were tested and found to be well within the minimum requirements. (See Table VIII.) It was also attempted to lessen the effect by diffusion annealing, but these specimens were of a lower strength than those originally tested indicating that the surface nickel content was excessive.

3.6 DIFFUSIVITY CALCULATIONS

Based on available diffusion data, the extent of nickel diffusion noted in these extrusions would not be expected. Calculations of the expected depth of diffusion based on theory are shown below:

In the calculation of the depth of nickel diffusion into the PH 15-7 Mo Steel extrusions the following assumptions were made:

- Nickel content in excess of 9 percent will cause excessive retained austenite.

- The diffusion coefficient of nickel into PH 15-7 Mo is essentially the same as that for nickel diffusion in pure iron.

- The diffusion coefficient of nickel into pure iron is described by the following equation:

$$D = 0.77 \exp - \frac{67{,}000}{RT} \quad *$$

Where: R = Universal gas constant
T = Absolute temperature

* Diffusion of Nickel into Iron

K. Hirano, M. Cohen, B. L. Averbach
ACTA Metallurgica, Vol. 9, May 1961

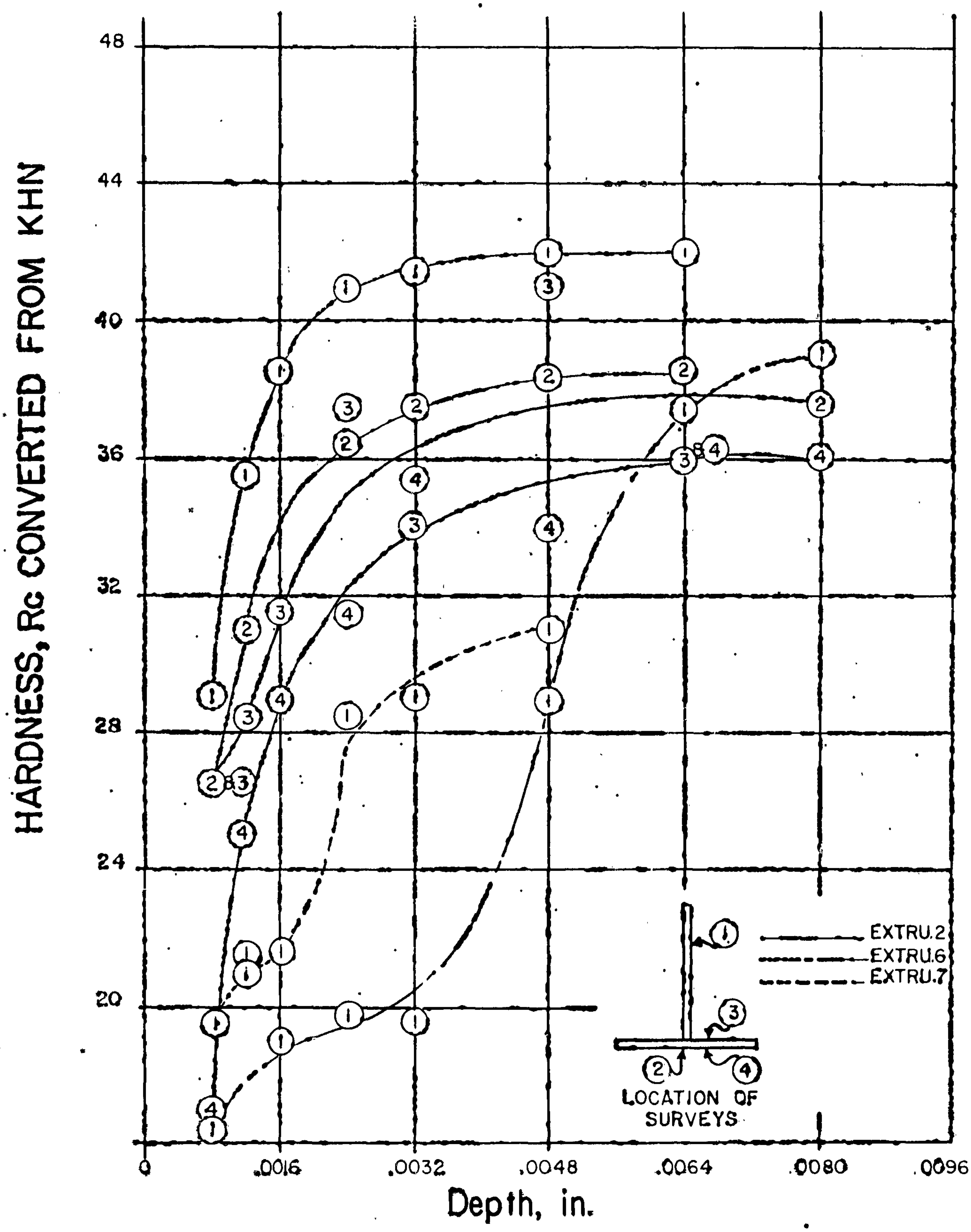

FIGURE 4. HARDNESS vs Depth in PH 15-7Mo EXTRUSIONS

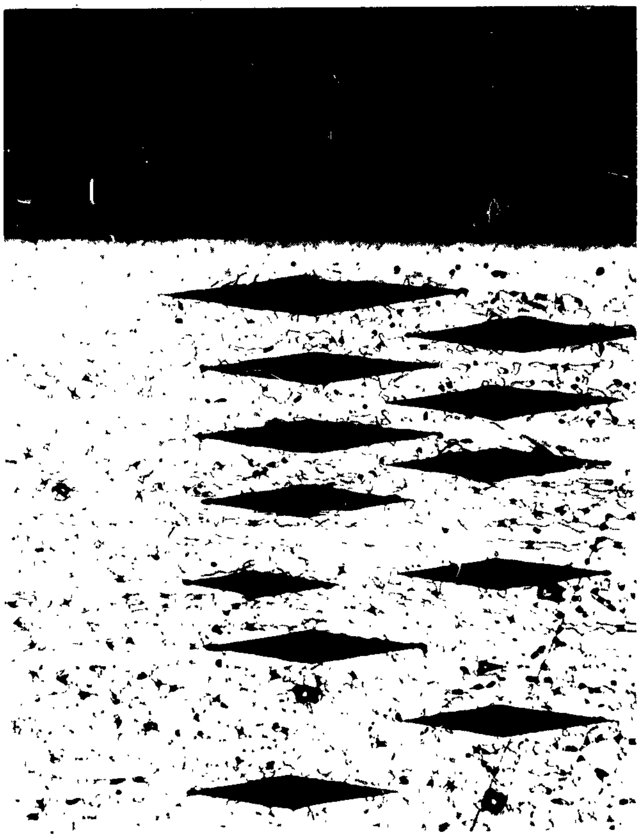

FIGURE 5 10% Oxalic Acid Electrical Etch -
Photo Micrograph of Hardness Traverse

TABLE VIII

TEST RESULTS TO DETERMINE STRENGTH

WITHOUT NICKEL DIFFUSED LAYER

Extrusion No. 6 (PH 15-7 Mo)

All Specimens taken longitudinal

0.010 in. material machined from each surface

Specimen Number	Yield Strength ksi	Ultimate Strength ksi	Elongation in 1 in. %
1	196.2	220.3	10.5
2	181.1	221.1	8.0
3	153.7*	221.9	11.0
4	191.7	222.8	10.0

* Value doubtful due to extensometer difficulties.

Nickel stripped from surface and extrusion homogenized at 2200F for 2 hr., then annealed at 1950F for 1 hr.

Specimen Number	Yield Strength ksi	Ultimate Strength ksi	Elongation in.1 in. %
1	161.5	213.3	5.0
2	145.4	205.4	12.0
3	160.3	212.4	8.0
4	175.6	212.4	12.5

All specimens austenite condition annealed at 1750F for 10 min., sub-zero cooled to -100F for 8 hr. and aged at 950F for 90 min.

- The equation describing the diffusion process and boundry conditions is as follows:

$$\frac{C_x - C_o}{C_s - C_o} = 1 - \text{erf}\ \frac{x}{2\sqrt{Dt}}$$

Where: C_x = Solute concentration at depth
C_o = Original uniform solute concentration
C_s = Surface concentration of solute
x = Distance from surface at which concentration C_x occurs after time t
t = Time at temperature
D = Diffusion coefficient.

$$\text{erf}\ z = \frac{2}{\sqrt{\pi}} \int_0^z e^{-z^2}\, dz$$

<u>Calculation:</u>

1. Diffusion occuring during billet heating.

$C_x = 9\%$ $\qquad t = 2\ \text{hr}$

$C_o = 7\%$ $\qquad T = 2200°F$

$C_s = 100\%$

$$D = 0.77e^{-\frac{67{,}000}{RT}}$$

$$D = 9.368 \times 10^{-11}\ \text{cm}^2/\text{sec.}$$

$$\frac{C_x - C_o}{C_s - C_o} = \frac{9-7}{100-7} = 1 - \text{erf}\ \frac{x}{2\sqrt{Dt}}$$

$$\text{erf}\ \frac{x}{2\sqrt{Dt}} = 0.9785$$

$$\frac{x}{2\sqrt{Dt}} = 1.626$$

or $x = 2.671 \times 10^{-3}$ cm
or $x = 1.052 \times 10^{-3}$ in.

2. Diffusion occuring during annealing.

$C_x = 9\%$ $\quad t = 1.5$ hr

$C_o = 7\%$ $\quad T = 1950°F$

$C_s = 100\%$ $\quad D = 8.934 \times 10^{-12}$ cm^2/sec

$$\frac{x}{2\sqrt{Dt}} = 1.626$$

$$x = 7.141 \times 10^{-4} \text{ cm} \quad \text{or} \quad 2.811 \times 10^{-4} \text{ in}$$

3. Thickness of diffused layer in final extrusion:

Volume of nickel contaminated material in billet

= .1011 in^3

Assuming that the entire billet was extruded the contamined layer would

= 4.5×10^{-5} in.

The additional diffusion from annealing from above

= 2.811×10^{-4} in.

Total thickness of diffused material in the extrusion = 3.261×10^{-4} in.

The above analysis indicates that theoretically the diffusion of nickel should be very slight. It was, therefore, never taken into account. However, it is apparent that the diffusion rate under the dynamic conditions of the extruding process is influenced greatly. The rate may either be accelerated by the extreme pressures involved in extruding, or by the severe deformations involved or a combination of both. The exact mechanism of the phenomenon observed is not important, however. This diffusion or contamination must be eliminated. It should be noted that for many extrusion applications this condition would not be serious; however, in this case where the thickness is only .06 the effect is of serious consequence.

Figure 6 is a photomicrograph of the material at the surface of one of the PH 15-7 Mo extrusions. The retained austenite is readily apparent in the microstructure. The white layer at the extreme top is the pure nickel coating. Note also the amount of delta ferrite present in the material. It is believed that this ferrite is responsible for the tearing occuring when extruding is attempted without cladding. It is therefore, considered doubtful that extruding of the PH 15-7 Mo can be accomplished without the nickel clad.

Assuming that the diffused area must be eliminated, the remaining problem is that of ascertaining which method will be the best means of accomplishment: either eliminating the nickel or secondarily providing a means of preventing the diffusion of the nickel into the billet material. Both approaches will be tried in forthcoming extruding efforts from the standpoint of solving the problem and from the aspect of improving producibility.

It should be noted, however, that preliminary tests on chemical milling extruded PH 15-7 Mo show promise. Substantial precentages of the thicknesses are removed in a short time and the resultant surface is appreciably smoother even than the original extruded surface. This improved condition obtained by chemical milling may very well be of substantial benefit in the cold and warm drawing operation. Another potential benefit of utilizing chemical milling would be that it would allow the thickness of the extrusion to be .09 or .10 which would be much easier to extrude than .06.

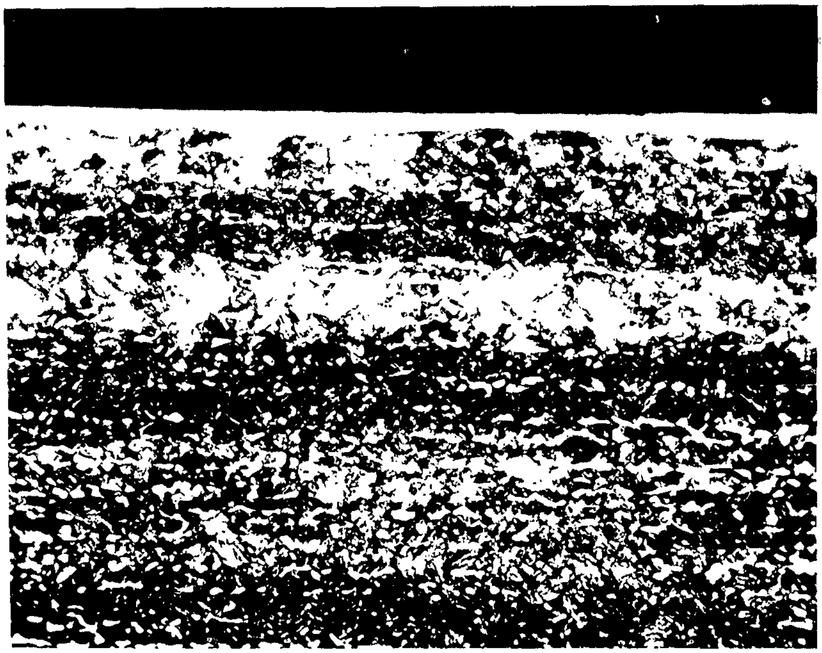

FIGURE 6 Mag-250X Marbles Etch
Photomicrograph Showing Retained Austenite in PH15-7MO Extrusion

4.0 PHASE II COLD AND WARM DRAW DEVELOPMENT

PROGRAM PLANNING

Upon receipt of the revised contract incorporating cold and warm draw techniques, details of the various areas of investigation were finalized. The following is an outline of the program plan.

Extruding

To provide stock for the draw experimentation and to improve the producibility of the extruding process. The following extruding effort is planned in Phase II.

1. Heating Study

   The salt bath which had been used for Phase I development has since been replaced by induction heating. Since it is necessary to heat the small billets involved in this program in a radiation shield, the practicability of utilizing this new method is in serious question. Consequently the various methods of heating will be re-analyzed and followed by tests with thermocouple probes in the billet to determine the heating patterns.

2. Contamination Study

   Reasons must be determined for substandard mechanical properties obtained in Phase I extrusions. Methods must be devised for elevating properties to desired levels and tests must be performed as necessary to substantiate reliability. Section 3.0 of this report gives full details of this study.

3. Die Material

   Inco 713C nickel base alloy dies will be evaluated and results compared with the presently used cobalt-base dies.

4. Fillet Radius Reduction

   Attempts will be made during the initial extruding effort to reduce the size of the extruded fillet radius from the present .13 to approach the .06 R which is desired in the drawn section.

5. Bare Extruding

   To solve the contamination problem and to economize the product, bare billets of both H-11 and PH 15-7 Mo will be attempted under the latest extruding technology. This will be compared with the present product that utilizes billets with an .04 thick coating of pure plated nickel.

6. Nickel Sheet Cladding

To eliminate the contamination problem, billets will also be tried with a sheet of nickel wrapped over the billet. The billet will receive a conversion coating prior to encompassing with the sheet to provide a barrier against diffusion of the nickel into the parent material.

7. Comparison of PH 15-7 Mo Heats

In recogniation of the ferrite problem which sometimes exists with PH 15-7 Mo, billets of both the newly procured Armco heat and from the same consutrode heat which was used in Phase I will be extruded and compared.

Post Extruding

1. Deglassing and cleaning
2. Vacuum annealing
3. Straightening
4. Transfer to cold and warm draw

Cold and Warm Draw

1. Review of Available Literature

A complete literature review will be made of lubricants for cold and warm drawing die materials, and die designs. Contact will be made with other groups with similar experience to obtain a composite of information for the best direction of this program.

2. Evaluation of Warm Drawing Facilities

a. The necessary 100 kilowatt heater, induction coils, temperature sensing and control equipment must be obtained, set up and standardized. This will permit drawing material from room temperature up to 1300°F.

b. Drawbenches are to be modified substantially. Gripping or shapes must be vastly improved. Die allignment facilities must be added. Guides must be added to prevent warping during drawing. Load measuring facilities must be added.

c. Pointing Facilities - Determination must be made of best means of pointing extrusions - grinding, pickling, and roll forging.

3. Lubricants and Surface Coatings

a. Surface Preparations to be Evaluated - Grit blasted surfaces, oxalate coatings, sulfur treated surfaces, and others as determined by literature search.

b. Cold Lubricants - Standard dry soaps, magnus 400, steel skin, silicates, lime, chlorinated oils, J-2, diegard, graphite, molydisulphide, teflon with volatile carriers, greases and waxes fortified with solids.

c. Warm Lubricants - In addition to several lubricants listed in above item b. shall be hot rolling oils such as Haas D200-50 and glasses with softening points from 750°F to 1300°F.

4. Dies to be Evaluated

a. Die material

(1) Carbide
(2) Standard tool steel (cold draw only)

b. Die design

(1) Solid dies
(2) Split dies
(3) Built-up dies in holder

c. Die contour

(1) Bearing length variation
(2) Approach angles; number angle and blend

5. Heat Treatments for Drawing

a. Annealed condition
b. Highly tempered hartensite

6. Variation of Drawing Parameters with Constant Lubrication Techniques at Temperature

a. Draw speed - 6 to 36 ft/min
b. Reduction per pass
c. Multiple passes between anneals
d. Record draw force for each condition

5.0 PROGRAM FOR NEXT QUARTER

During the months of July, August, and September 1961, the following is expected to be accomplished:

- Preliminary lubrication tests utilizing the wide variety of contemplated lubricants will be tried under simulated conditions on strips of PH 15-7 Mo sheet. This will be an economical way to eliminate the less promising lubes. The balance will then be utilized under actual operating conditions.

- Both H-11 and PH 15-7 Mo billets will be extruded under various extruding parameters to furnish extrusion stock for drawing experiments, to refine the technique for producibility reasons, and to eliminate the nickel diffusion problem which has resulted in lowered properties.

- The induction heating unit which has been fabricated to preheat the extruded shape as it goes into the draw die aperture will be sent to Allegheny. It will then be set up and calibrated with the temperature sensing and control equipment. This will include revamping Allegheny's extruding plant main electrical system.

- The draw bench on which the cold and warm drawing will be accomplished will be altered and equipped with the induction heater, new gripping jaws, guides, improved alignment facilities, and force recording equipment.

- The draw dies which have been designed and ordered will be procured, set up, and cold and warm draw experiments will be started under various conditions of lube, temperature, heat treat condition, etc.

APPENDIX I

Summary of previous Interim Reports published under Contract AF 33(600)-36713

Interim Engineering Report No. 1, NAI-58-656, included a full discussion on the surveys of both the airframe and the steel extruding industries. Also included was the basis for selecting the Phase I shape, the target specifications, and the initial three participating subcontractors.

Interim Engineering Report No. 2, NAI-58-876, included a full discussion of Allegheny Ludlum's equipment, extruding precepts, and facilities, as well as reporting the first half of their experimental extruding effort. It covered only the preliminary large diameter container pushes at Harvey Aluminum because at the close of the report period their small diameter container had not been completely fabricated. Interim Report No. 2 did not include any technical data from C. I. E. P. M. because Sejournet's extruding effort did not begin until after the issuance of that report.

Interim Engineering Report No. 3, NOR-59-246, included a detailed description of the last half of Allegheny Ludlum's effort since the first portion was covered in Report No. 2. The extruding effort by C. I. E. P. M. (Sejournet) was included in its entirety with extensive inspection data obtained from their several extrusions. No coverage was given to Harvey Aluminum's effort because Harvey was late in submitting their report.

Interim Engineering Report No. 4, NAI-59-354, included a complete tabulation of detailed inspection and evaluation of samples submitted from each of the three participants. Such items were listed as dimensional variation, surface roughness, and inclusion count. Also included was the complete presentation of the entire Phase I effort for Harvey Aluminum. A discussion and summation of the scheduled Phase I effort was presented together with details of an extended development effort for Phase I with Allegheny Ludlum as subcontractor.

Interim Engineering Report No. 5, NOR-59-504, included details of the design and utilization of a radiation shield for heating and handling of billets. Also included was information and technical substantiation for a high-heat container liner which was conceived and fabricated primarily for extruding A-286 material. The report presented details of an extruding effort with the high-heat liner which was foreshortened due to premature container and stem failure. Efforts to replace these failed major components was underway when the steel strike halted all activity,

Interim Engineering Report No. 6, NOR-60-106 includes activities starting from the end of the steel strike on 7 November 1959. After resumption of work schedules, some time was consumed in reactivating the plant and facilities during which a new liner and new stems were being finished. Extruding of A-286 was again attempted with the high heat liner and again resulted in gross failure of tooling. Subsequently, PH 15-7 Mo was substituted for A-286. Another extruding effort was made with H-11 and the new PH 15-7 Mo using a container liner at the more conventional 900°F temperature. It is anticipated that extruding of A-286 will possibly be resumed at a later stage in the program.

Interim Engineering Report No. 7, NOR-60-253, completely summarized Phase I effort. A major extruding effort was planned and effected using H-11 and PH 15-7 Mo. A new stable die material was used extensively which, in the previous period, had indicated promise. A new higher melting point glass was used with billets that received a longer soak period at temperature. Results were deemed to be an unprecedented success in efforts to produce super thin (.06) thicknesses of aircraft type shapes in difficult-to-form alloys.

APPENDIX II

Subject: PH 15-7 Mo Extrusion - Norair Division of Northrop Corporation

S. I. 61-349

An extrusion of PH 15-7 Mo approximately 14 inches in length was received from Norair Division of Northrop Corporation. The other dimensions of the extrusion were:

Flange - .070" x 1 1/2"

Stem - .065" x 1 1/2"

It is understood that Norair has a contract from Wright Field to study the feasibility of producing thin wall extrusions. This extrusion was produced by Allegheny-Ludlum under subcontract with Norair. The temperatures were reported to be 2175°F with a reduction ratio of approximately 20 or 30:1.

North American Aviation has examined these thin wall extrusions and concluded that they are not acceptable. Research was asked to examine the extrusion and comment.

CHEMICAL ANALYSES

The "as received" extrusion had a nickel clad surface .001 to .002" thick over which plastic had been sprayed. The nickel was removed in boiling nitric acid prior to making the chemical determinations at Research. The analysis by direct reading spectrograph by Norair and the check analysis made at Research follow:

	C	Mn	Si	Cr	Ni	Cu	Mo	Al
Spectro	---	.58	.42	15.65	7.34	.14	2.46	1.32
Research	.06	---	---	15.45	7.42	---	2.16	---

The chromium, nickel and aluminum are within specification, but they are on the high side of the range.

MECHANICAL PROPERTIES

The mechanical properties were not determined at Research. Norair had evaluated the mechanical properties much more thoroughly than we could do it with the small sample that was submitted.

Sample 2AM-3 has a very low tensile and yield strength. With the exception of this sample, the series 2 specimens had a range in yield strength of 190,600 to 212,700 psi and a tensile strength of 200,000 to 225,700 psi. The yield strength range for the series 6 specimens was 172,200 to 206,600 psi and for the tensile strength it was 196,000 to 216,700 psi. With the exception of one sample, none of these specimens met Condition RH 950 strength properties for sheet and strip of this thickness. Information was not received as to whether the nickel coating was removed prior to testing. If not, then these results could be low by approximately 5%.

SURFACE EXAMINATION

The extrusion had a number of defects on the surface of the flange and to a lesser extent on the stem. These defects are shown in Figure 1. The notches formed by these defects could cause failure of the part if subjected to fatigue of high load stresses. Sufficient machining to remove the defects would be necessary.

MICROSCOPIC EXAMINATION

Microscopic examination was made of a section that had been annealed and of one that was annealed and heat treated to Condition R-100. In Figure 2, the microstructure of the heat treated piece is shown at one surface of the stem. The nickel coating is out of focus due to etching the sample. A nickel diffusion layer can be clearly seen at the surface. Very slight differences in etching characteristics can be seen under the nickel layer at the right of the micrograph.

Figure 3 is a photomicrograph taken at the radius at the intersection of the flange and stem. Again the nickel layer as well as the distinct diffusion layer is out of focus due to attack from the etchant. However, the light etching area immediately under the surface is broader and can be seen more readily than in Figure 2. Hardness of the area and of the center of the extrusion follows:

Edge of Extrusion		Center of Extrusion	
DPH	Rockwell (converted)	DPH	Rockwell (converted)
187	90.5B	333	34.0C
266	25.5C	346	35.5C
211	95.0B	297	28.0C
225	97.5B	297	28.0C

The above values show that the slight etching area under the surface is definitely softer than the dark etching area at the center of the sample. This would indicate that austenite is responsible for the lower hardness of the area under the surface. The austenite may be due to nickel diffusion into the base metal during heat treatment. However, this cannot be stated positively.

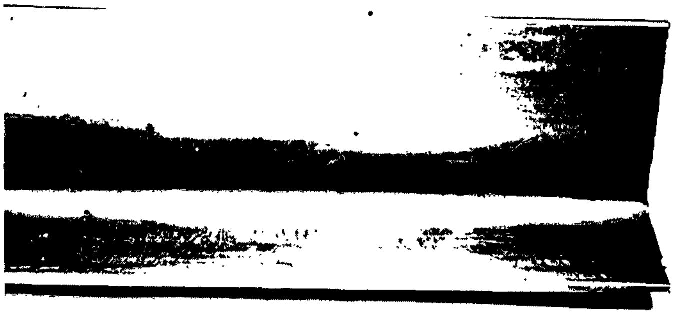

Figure 1 M61251

Surface defects on flange and stem of extruded "T" section.

Mag. 2/3 x

Figure 2 M61252

Mag. 500X Etch-HNO Ac, Electrolytic

Nickel coating etched and out of focus. PH 15-7 Mo base metal showing layer at surface where nickel has diffused.

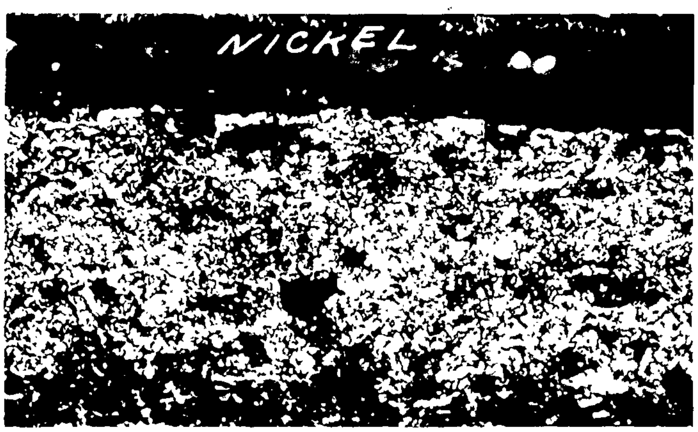

Figure 3 M 61253

Mag. 500X Etch-HNO Ac, Electrolytic

Light etching area of low hardness at surface of extrusion. Area appears to have retained austenite which may be due to nickel diffusion. (Cause not determined).

CONCLUSIONS

The surface of the extrusion received for examination would require machining or grinding to remove the defects. The surface quality is not acceptable as extruded.

The nickel cladding or plating should be removed prior to annealing and heat treating. Some diffusion has occurred into the base metal, although we cannot definitely determine the full extent of the diffusion.

The mechanical properties obtained were not the equivalent of these obtained with sheet and strip. The decision as to whether the properties were acceptable would depend on the requirements for the part and the reproducibility of properties from extrusion to extrusion.

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